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(6) ADVANCED FUEL SYSTEMS FOR RAMJET-POWERED VEHICLES.

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1.0 Summary

The major efforts during this reporting period were in completing the sample preparation for all the fuel compatability screening tests, and in completing the fuel characterization work. The remainder of projected Shelldyne H requirements for the program were delivered during this month.

A total of eight metals, five elastomers, and three plastics have now been placed in Shelldyne H at both room temperature and at elevated temperature for immersion compatability testing. Preliminary results include severe discoloration of the fuel by ethylene propylene terpolymer. The five elastomers have been placed in permeability testing, with early indications that the Shelldyne H permeates and distorts ethylene propylene terpolymer. The eight metals have been placed in stress-corrosion (U-bend) testing.

The fuel characterization and storability effort included an existent gum determination, an oxidation stability test, another particulate contamination measurement, and a bromine number determination. Unused Shelldyne H has turned a distinct yellow in color, and the fuel subjected to the oxidation stability test has turned a darker yellow. The high particulate contamination level reported last month was apparently due to an extranneous introduction of foreign material, since a repeat measured this month revealed negligible contamination.

Some additional efforts were made during this month in detailing the revised high-shear apparatus design, and in more closely defining the specimen configuration for the pressurant compatability tests.

2.0 Fuel Compatability - Screening Tests

During this reporting period, all of the sample preparation was completed for the immersion tests, for the U-bend stress corrosion tests, and for the permeability tests. Figure 2.1 lists the materials selected and subjected to these screening tests. These include eight metals (four steels, two aluminums, and two titaniums), five elastomers, and three plastics. One additional metal, aluminum 2219, has not as yet been received from the vendor.

2.1 Immersion Tests

A sufficient number of samples of each material are immersed in Shelldyne H, both at room temperature and at 160°F for accelerated testing.

Figure 2.1

Candidate Materials for Screening Tests

Material	Immersion	Stress Corrosion	Permeability
Metals:			
Steel 4130	X	X	
Cadmium - Plated Steel 4130	X	X	
Stainless Steel 321	X	X	
Stainless Steel 14-8	X	X	
Aluminum 7075	X	X	
Aluminum 6061	X	X	
Titanium 6 Al-4V	X	X	
Titanium 5 Al-2.5 Sn	X	X	
Bladder Candidates:			
Viton (US 3094)	X		X
Viton/Nomex (US 941)	X		X
Ethylene Propylene Terpolymer (US 3015)	X		X
Nitrile (US 3010)	X		X
Nitrile/Nylon (US566RL)	X		X
Plastics:			
Nylon	X		
Teflon	X		
Silicone	X		

The attached photographs (Figures 2.2 and 2.3) show the immersed samples at room temperature.

The initial screening phase of Task I includes the visual examination of both the specimen and the fuel at two week intervals. The fuel, both at room temperature and at 160°F, has been heavily discolored to yellow by the ethylene propylene terpolymer and lightly discolored by the nitrile, the nitrile/nylon, the viton and the viton/nomex. Rust has attacked both the 4130 steel control coupons at room temperature and room temperature Shelldyne H. No rust can be seen on the 4130 coupons in 160°F Shelldyne.

2.2 Permeability Tests

Duplicate samples of the five candidate bladder materials are under test for permeability to Shelldyne H. A photograph (Figure 2.4) shows the permeability tests with Payne cups. While several weightings have been made and weight losses determined, the permeability will not be determined until the cups reach equilibrium. It should be noted that the ethylene propylene terpolymer (3015) quickly distorted and is wet through with the fuel.

2.3 Stress Corrosion Tests

Stress corrosion tests are under way with all the metals. A U-bend specimen of each is immersed in Shelldyne H at room temperature.

3.0 Fuel Characterization and Storability

During this reporting period, 282 gallons of Shelldyne H, from Shell Development Company, Lot 10704-67A (dated 5 August 1969), were received. This shipment, plus the 10 gallons previously received (Lot 10704-45, dated 7 July 1969), complete the projected Shelldyne H requirements for this program.

During this reporting period, the physical and chemical characterization of the "as-received" fuel was completed. Sufficient data has now been recorded to satisfy the objectives of fuel data for ramjet design and analysis, and of initial fuel properties for later comparison with stored fuel or with fuel in contact with ramjet components materials.

The results of density, viscosity, vapor pressure, particulate contamination, and acid number measurements, and a chromatogram and an infrared spectra, were reported in the previous monthly report. The following results were obtained during the month of August 1969:

12006-4
23155

Figure 2.2. Room Temperature Immersion Tests.

12306-3
23156

Figure 2.3. Room Temperature Immersion Tests.

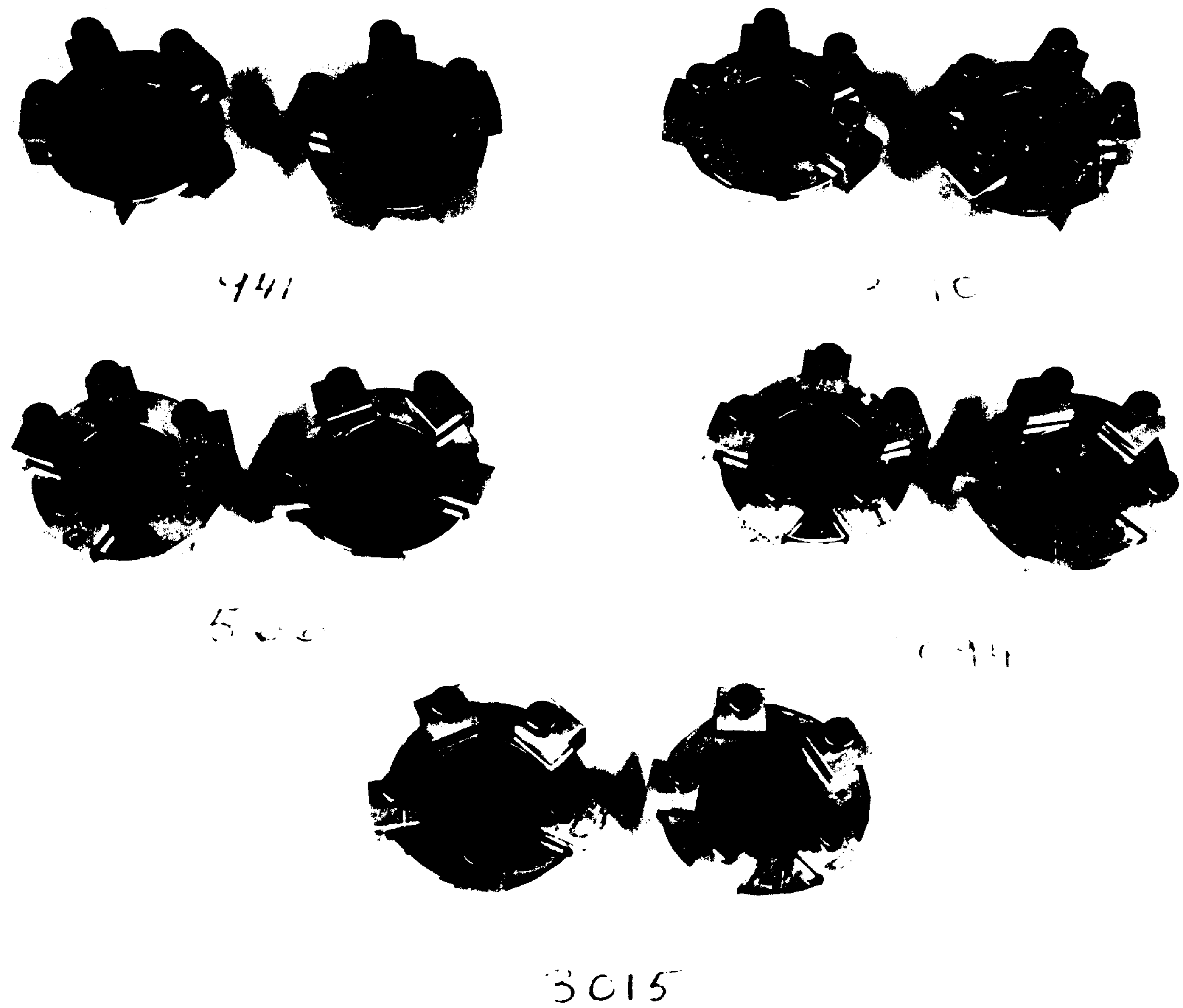

Figure 2.4. Permeability Tests.

12006-1
23157

3.1 Existent Gum (ASTM D381)

A sample from Lot 10704-45 indicated 5.4 milligrams per 100 ml. This is within the maximum of 7.0 mg/100 ml of the proposed Military Specification (dated February 1969).

3.2 Oxidation Stability

Dried air at a rate of 5 liters per hour was bubbled through 300 ml of 160°F Shelldyne H (Lot 10704-45) for 48 hours. After cooling, a sample of the fuel was mixed with petroleum ether. No insoluble decomposition products were observed.

However, the Shelldyne H had turned a distinct yellow color as the result of this treatment. It has also been observed that the fuel of Lot 10704-45, which has been stored in the original (opened) container, has become a light (but distinct) yellow in color. This aging characteristic will be studied more closely throughout the program.

3.3 Particulate Contamination (ASTM D-2276)

Because of the high value of 40 milligrams per liter measured during July, another determination was made this month. This time, the measured level was less than one milligram per liter, well within the proposed Mil Spec maximum value of 2.0 mg/liter. A light gray smudge visible on the test millipore filter was of negligible weight.

The discrepancy between the two measurements might have these explanations:

a. The July measurement was made by Penniman and Browne, Inc., Baltimore, Maryland, as subcontractors to Atlantic Research Corporation; while the August measurement was made at Atlantic Research Corporation. Foreign material could conceivably have been introduced into the July sample as it was shipped from ARC to Penniman and Browne.

b. Although both measurements were made from Lot 10704-45, they were made from samples taken from two different 5-gallon containers. Unfortunately, no fuel remains in the first container (from which the July data was obtained). It is possible that foreign material was introduced into this container either during packaging or after opening at ARC.

As a result of the low contamination measurement of August, it is apparent that the high July level was the result of external contamination rather than the result of any fuel degradation. It is therefore concluded that the July value was anomalous (regardless of its source), and should be disregarded.

3.4 Bromine Number (ASTM D-1158)

The bromine number was determined for samples of both lots of Shell-dyne H:

Lot 10705-45	Bromine Number = 3.3
Lot 10704-67A	Bromine Number = 2.8

During these determinations, it was observed that the addition reaction of bromine to residual double bonds proceeded slowly. This was evidenced by the back-titration of excess bromine with sodium thiosulfate, which required fewer milliequivalents of $Na_2S_2O_3$ than the excess KBr-$KBrO_3$ (added after the first 5-second appearance of a yellow color). Hence, the bromine number as determined above would be greater than the bromine number as determined by direct electrometric titration with KBr-$KBrO_3$ (ASTM D-1159), since the addition reaction was permitted to proceed further in the time prior to back-titration. These results, then, are not directly comparable to the proposed Mil Spec maximum value (for control of the hydrogenation process) of 1.0, using ASTM D-1159.

The method used, however, is indicative of the residual unsaturation in the fuel which could be subject to long-term oxidation or polymerization, or which could be reactive with ramjet fuel system materials. Changes with time of the bromine number determined by this method (ASTM D-1158) could be indicative of any changes in the fuel because of oxidation, polymerization, or reaction with other materials.

4.0 High-Shear Apparatus Design Revisions

The detail design work for the revisions outlined in the previous report are 70 per cent complete. Since the redesign concept satisfied the technical requirements of the program, the detailing work has been paced to meet the scheduled use of the equipment in December 1969.

5.0 Gas Generator Test Motor Specimen Configuration

The design of the gas generator test motor, described in the previous report, was purposely made flexible to accomodate a wide variety of specimen configurations.

As originally conceived, strips of candidate bladder materials would be exposed to 2200°F gases. The analysis reported last month showed that this was much too severe (and unrealistic) a test, since hot gas would be continuously supplied and since there would be no back-cooling from fuel.

A simple alternate and more meaningful scheme uses two one-inch stainless steel Swagelok tube-fitting crosses, each of which to be filled with Shelldyne H. The four openings of each cross will each accomodate a one-inch disc of elastomer, sealed between two flat phenolic washers held by the Swagelok nut. A total of eight elastomer specimens can then be tested at once, using the two independent fuel chambers. The crosses will be insulated on the outside, and supported by an axial bolt welded to the face of the fittings and which screws into the aft closure plate of the test motor.

6.0 Work Planned for Following Period

6.1 Obtain quantitative results from fuel compatability tests. Immersion coupons will be pulled and analyzed, fuel will be analyzed, permeability rates will be measured.

6.2 Design of special test equipment for Task II will begin.

6.3 Gas generator test motor will be fabricated.

6.4 High shear apparatus will be revised.